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13. ABSTRACT *(Maximum 200 words)*

We have completed etch rate determination and plasma diagnostics using optical emission spectroscopy, mass spectrometry, and microwave measurements of average electron densities. The previous diagnostics indicate that the observed etch rate enhancement might be due to in part to an increase in the overall electron density (but not enough to account for the entire etch rate enhancement) and a probable increase in the average electron temperature (energy), both of which lead to increased dissociation of SF6 and the creation of more etch species. We have finally obtained reproducible Langmuir probe results, which show that the electron temperature does indeed increase with the addition of helium to SF6.

The design of the Langmuir probe and the measurement technique have been discussed in detail in previous reports. We have found that the key to obtaining reproducible measurements ties with the cleaning procedure used between measurements. This cleaning . procedure removes any impurities that may have collected on the probe tip. This procedure consists of driving the probe tip to carry a high current in inert plasmas (e.g., argon or helium) in order to heat the probe tip to induce refractory cleaning. Plasma chemistries containing SF6 are notorious for contaminating probe tips. The cleaning procedure . we have developed which gives reproducible results is given, on the next page.

14. SUBJECT TERMS

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(Final) **Performance Report**

"Characterization of Plasma Etch Processes for Wide Bandgap Semiconductors"

DEPSCoR FY02, Award Number F49620-02-1-0220

September 7, 2005

Principal Investigator: Karen J. Nordheden

Institution: University of Kansas
C&PE Department
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Objectives:

The overall objective of this work is to develop and characterize plasma etch processes for wide bandgap semiconductors. The original proposal included the optimization of SF_6 plasmas to etch SiC described in 1) below. Since the time the grant was awarded, we have expanded the original proposal to include 2) which is a collaboration with Michael Alexander and Michael Callahan at Hanscom AFRL and Ferechteh Teherani at Nanovation to attempt to etch ZnO and to see if there are crystallographic dependencies on the etch rate.

1. Optimization of Silicon Carbide plasma etch processes:

The major component of this research is to characterize sulfur hexafluoride (SF_6) gas mixtures to etch silicon carbide. The addition of argon or helium in the gas mixture will be studied to determine the effects on etch rate and surface morphology. Characterization of these plasmas is necessary to fully understand the complexities of the etch mechanisms. The plasma diagnostic tools we will be using include mass spectrometry, optical emission spectroscopy, and Langmuir probe and microwave measurements of the electron density. The results of the plasma characterization experiments will then be used to optimize the etch rate, etch selectivity, anisotropy, and surface morphology, in order to ultimately enhance device performance.

2. ZnO Plasma Etching:

This is a new objective not included in the original proposal. The only samples we have obtained to date are bulk ZnO samples from two different boules. A review of the literature revealed that very little is known about dry etching of ZnO (there are about 5 articles in total). Chlorine and methane based chemistries have been used primarily, and all the efforts have used Inductively Coupled Plasma (ICP) systems. For the time being, our research must be done in our current system which is an RIE. We expect that our rates will be much lower than those obtained in an ICP.

Status of Effort:

Previously, we reported the results of our research on SiC etch rates, optical emission spectroscopy, and microwave measurements of electron density in SF_6/He mixtures. We found that the observed enhanced SiC etch rate at a pressure of 125 mTorr and a composition of 50% SF_6 in helium appeared to be the result of both an increase in the overall electron density and a probable increase in the electron temperature. Since the time of our last performance report (September 2004), we have continued our work to develop reproducible electron temperature (energy) measurements. After finally developing a reliable cleaning procedure, which is implemented between measurements, our results are now reproducible. We have found that there definitely is a marked increase in the electron temperature with the addition of helium to SF_6. Furthermore, the electron temperature we measured for pure helium is within the range of published data for RIE systems.

Our work on ZnO etching remains hampered by the fact that we do not have easy access to an ICP system (KU doesn't have one). However, we have been able to arrange for two bulk samples to be etched in an ICP using BCl_3/CH_4 gas chemistry with good results. The etch rate for these initial runs was between 1250 Å/min and 1400 Å/min. It also appears that the university (KU) is going to invest in the purchase of an ICP system to be located in our new multidisciplinary research building (which will be completed near the end of this year). This ICP system will be available to our group to develop etch processes for researchers across campus.

Accomplishments/New Findings:

1. **<u>Optimization of Silicon Carbide plasma etch processes:</u>**

We have completed etch rate determination and plasma diagnostics using optical emission spectroscopy, mass spectrometry, and microwave measurements of average electron densities. The previous diagnostics indicate that the observed etch rate enhancement might be due to in part to an increase in the overall electron density (but not enough to account for the entire etch rate enhancement) and a probable increase in the average electron temperature (energy), both of which lead to increased dissociation of SF_6 and the creation of more etch species. We have finally obtained reproducible Langmuir probe results, which show that the electron temperature does indeed increase with the addition of helium to SF_6.

The design of the Langmuir probe and the measurement technique have been discussed in detail in previous reports. We have found that the key to obtaining reproducible measurements lies with the cleaning procedure used between measurements. This cleaning procedure removes any impurities that may have collected on the probe tip. This procedure consists of driving the probe tip to carry a high current in inert plasmas (e.g., argon or helium) in order to heat the probe tip to induce refractory cleaning. Plasma chemistries containing SF_6 are notorious for contaminating probe tips. The cleaning procedure we have developed which gives reproducible results is given on the next page.

Langmuir Probe Data Collection Procedure:

1) Pump down chamber
2) Bias Probe to 0V
3) Select Cleaning Pressure
    a. 75 mTorr for He
    b. 145 mTorr for Ar
4) Select Noble Gas and flowrate (20sccm)
5) Select RF Power
    a. 100W for He
    b. 50W for Ar
6) Select Time to Clean Probe (20 minutes)
7) Turn on MFC and begin to flow noble gas
8) Turn on Throttle Valve and wait for respective pressure
9) Turn on RF Power
10) Bias the probe to nominal current, while adjusting voltage to avoid plasma breakdown
    a. 55 mA @160V for He
    b. 50 mA @120V for Ar
11) Immediately after cleaning time has elapsed bias probe to 0V (See Step 6)
12) Wait 15 minutes for probe to cool down
13) Select Data Acquisition Pressure
14) Select Data Acquisition Gas Flow rates
15) Select Data Acquisition Power
16) Select Data Acquisition Time (>5 minutes, manually turn off)
17) Turn on MFC and begin to flow gas mixture
18) Turn on Throttle Valve and wait for respective pressure
19) Turn on RF Power
20) Wait for stable DCV in chamber (~30 seconds)
21) Begin Data Acquisition (Data sweeps below floating and above)
22) Record Chamber DCV
23) (Optional) Once Data Acquisition is Complete, repeat Steps 21 and 22 for additional runs at the same power, pressure, and gas percentages
24) Manually turn off power, pressure, and flow rate
25) Begin at Step 2 for another Data Acquisition Run at a different gas percentage

One example set of Langmuir probe results for a gas mixture of SF_6 and helium is shown in Figure 1 below. The data were taken at a pressure of 125 mTorr, a power of 175 W and a total flow rate of 20 sccm. The ascending and descending sweeps correspond to ascending voltage applied to the probe and descending voltage applied to the probe, respectively.

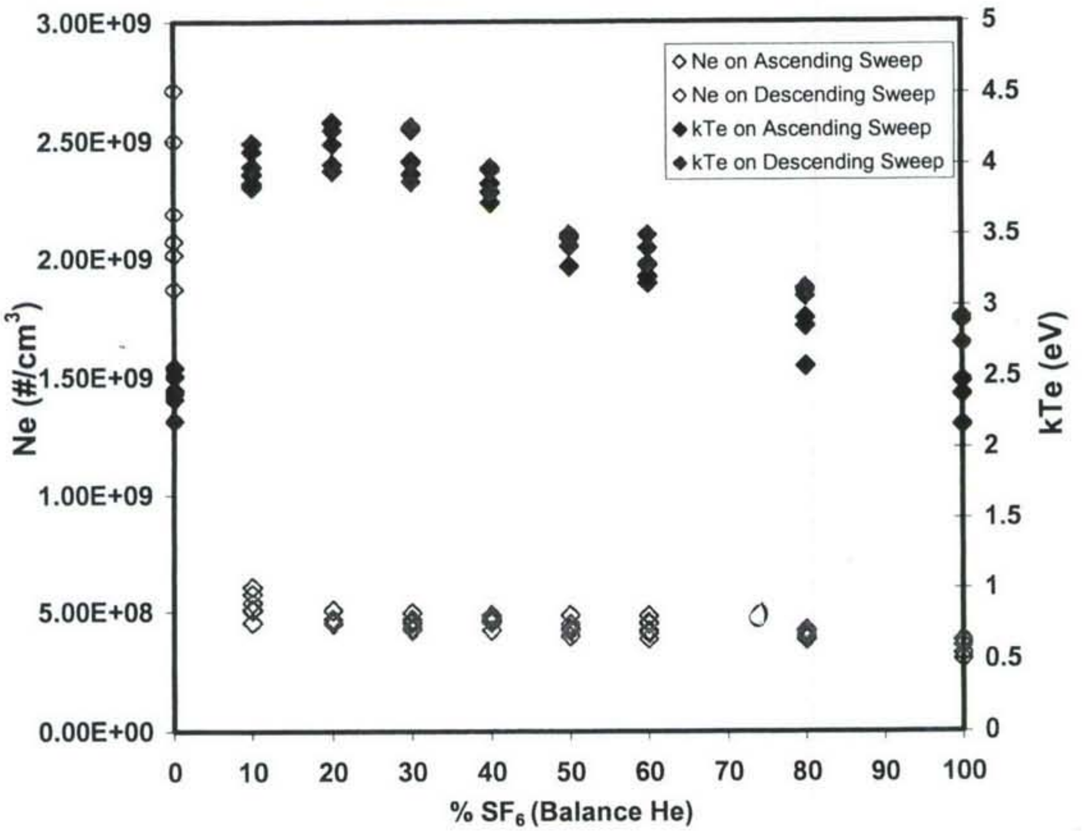

Figure 1. Electron temperature and electron density as a function of %SF_6 in He (125 mTorr, 175W and 20 sccm).

An average of three ascending runs under the same plasma conditions is shown in Figure 2 with error bars indicating the measurement variation. The data are very reproducible. **The average electron temperature increases from an average of 2.3 eV in pure SF_6 to a little above 4 eV in the 20 to 30 percent SF_6 range. This increase would significantly enhance the dissociation of SF_6 in the mixture leading to increased production of etch species and a higher etch rate.**

The electron density follows a similar trend to that observed using microwave measurements. The electron density is higher in pure He than in pure SF_6 and there is a slight increase in the density at 50% SF_6 from that of pure SF_6. The large decrease in

electron density with the addition of small percentages of SF_6 is most likely due to electron attachment.

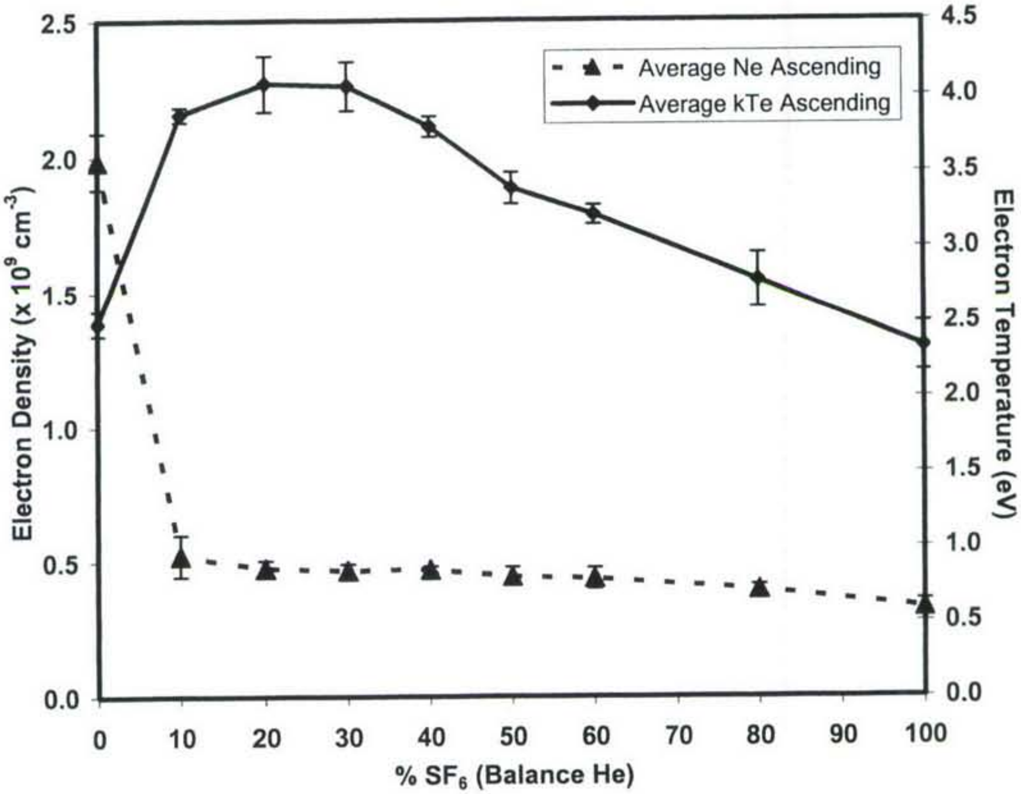

Figure 2. The electron density and electron temperature in SF_6/He gas mixtures averaged over three ascending runs (125 mTorr, 175W, 20 sccm).

As reported previously, the peak in the SiC etch rate in a SF_6/He plasma at a pressure of 125 mTorr and a power 175 W corresponds to 50% SF_6 as seen in Figure 3 on the next page. The discrepancy in SF_6 percentage between where the peak in the etch rate occurs (50% SF_6) and where the peak in the electron temperature occurs (near 25% SF_6) can be explained by the fact that as the electron temperature increases, the amount of SF_6 in the plasma is decreasing and thus there is less SF_6 to dissociate. It appears that the optimum condition for these two competing processes occurs at 50% SF_6 as indicated by the peak in the etch rate.

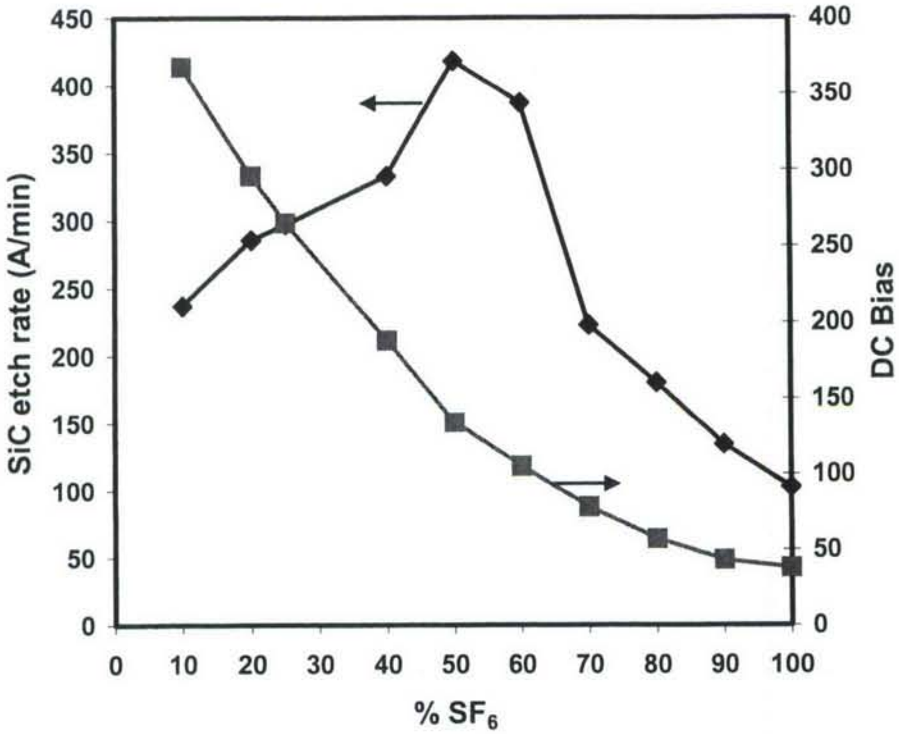

Figure 3: SiC etch rate as a function of SF_6 percentage (125 mTorr, 175 Watts, 20 sccm).

2. <u>ZnO Plasma Etching:</u>

Our work on ZnO etching remains hampered by the fact that we do not have easy access to an ICP system (KU doesn't own one). However, we have been able to arrange for two bulk samples to be etched in an ICP using BCl_3/CH_4 gas chemistry with good results. The conditions for the two etch runs were:

BCl_3/CH_4= 30 sccm/7.5 sccm (BCl_3:CH_4 is 4:1)
P = 5 mTorr
RIE = 350W
ICP = 1000W
DC bias = ~ 320 (-V)
Etch time = 5 min

The etch rates for these two samples ZNO41BH and ZNO41BG (HANSCOM) were 1250 Å/min and 1400 Å/min, respectively. The surfaces of both samples were smooth and clean. We are going to etch both samples simultaneously (in the chamber at the same time) with the same conditions above to determine if we are observing a true

difference in etch rate between the samples or whether the difference was just due to run to run inconsistency.

It appears that the university (KU) is going to invest in the purchase of an ICP system to be located in our new multidisciplinary research building (which will be completed near the end of this year). This ICP system will be available to our group to develop etch processes for researchers across campus. We hope to have this new system installed early next year. In addition, Ferechteh Teherani (Nanovation) has agreed to provide ZnO films for our etch studies, which will be easier to pattern than the bulk pieces for etch rate measurements. Since our current DEPSCoR grant has just expired, we plan to pursue additional funding in order to continue our ZnO etch studies.

Personnel Supported:

Along with myself, one graduate student (John Alexander, Chemical Engineering) is associated with this work. Bogdan Pathak (Electrical Engineering) is working on the Langmuir probe studies and was paid with internal KU funds.

Publications:

"Characterization of $SiCl_4/N_2$ Plasmas," A. S. Agarwal, V. Berry, R. Alapati, and K. J. Nordheden, J. Electrochem. Soc., **152**(3) G210-G212 (2005).

Future:

"Reactive Ion Etching of SiC in SF_6/He Plasmas," R. Alapati and K. J. Nordheden, still in draft stage (but now we have the electron temperature data to finalize the paper), Fall 2005.

"Advances in ZnO etching," K. J. Nordheden, SPIE conference proceedings 2006 (Invited).

Presentations:

None, but the SPIE talk mentioned above will be given in January 2006.

Interactions/Transitions:

We continue to keep in touch with Ferechteh Teherani (Nanovation), Mike Alexander (Hanscom AFRL), and Bish Ganguly (Wright Patterson AFRL).

New Discoveries, inventions, or patent disclosures:

None.

Honors/Awards:

None, unless the invited talk at SPIE counts.

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092632

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Progress on subject report covering the period from 01-May-2002 to 31-Aug-2005:

(✓) Received on 24-Mar-2006
(✓) Accepted on 27-Mar. 2006
( ) Nonacceptance-Contractor has been verbally informed of nonacceptance and a resubmission suspense date of _______________ is recommended. Attached is Principal Contracting Officer (PCO) letter to the Business Office and PI detailing reasons for nonacceptance and establishing a resubmittal date.

DON SILVERSMITH
Program Manager